

INTERIM REPORT 1 OF 2 CARBON DIOXIDE CONCENTRATION SYSTEM

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Prepared for

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ABSTRACT

13114

Under contract NAS 3-7638, the preliminary analysis has been completed for design, fabrication, and testing of a life support system utilizing the TRW-developed "Carbonation Cell" system of extracting CO₂ from cabin air.

Stages I and II of this Electrochemical Carbon Dioxide Concentration System have been designed and fabricated. The cells use electrodes measuring 12" by 12", and plated magnesium end plates.

Materials compatibility studies have also indicated the 2-mil non-porous gold plated magnesium is a suitable material to use in the third stage acid electrolyte cell. However, considering its prohibitive cost, plastic (PVDC) cells will be used for this program. These stage III cells are being designed.

A test rig for small cell testing was assembled and has been used for testing of small acid stage cells. Additionally, a small carbon cell and a gold plated magnesium cell are being fabricated and assembled for use in this test rig. Design of a large test stand is completed for testing the full scale electrochemical cells, and assembly and checkout also are in progress at this writing.

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SUMMARY

The objective of this contract is to obtain engineering data for an Electrochemical Carbon Dioxide Concentration System suitable to perform as a life support system in space. The system will consist of three connected electrochemical cells which extract CO₂ from air processed through the system.

A preliminary system analysis was completed to permit the subsequent design of the experimental cells (12" by 12" electrodes). First and second stage cell design and fabrication have been completed. The cell material is plated magnesium. The plating is underway at this writing.

The results of studies conducted to determine suitable materials for the acid electrolyte third stage cells indicated that 2-mil non-porous gold plated magnesium is the most suitable material for this purpose. However, its high cost is not compatible with the present program, hence, a plastic (PVDC) cell with 2-mil gold plated copper current collector inserts will be used for this experimental program.

A test rig for small cell testing was assembled and has been used for testing of a small plastic acid stage. Additionally, a small carbon cell and a 2-mil non-porous gold plated magnesium cell are being fabricated and assembled for use in this test rig. Design of a large test stand is completed for testing the full scale electrochemical cells, and assembly and checkout also are in progress at this writing.

1.0 INTRODUCTION

This is an Interim Report covering the first four months of work carried out at TRW Equipment Laboratories under contract NAS 3-7638. Under this contract, individual cells of a three-stage electrochemical carbon dioxide concentration system for space applications are to be designed, fabricated and tested. The electrochemical concept being used for carbon dioxide concentration was developed by TRW and the cells used in the system are called "Carbonation Cells". This system offers the following desirable features:

- 1. CO2 would be removed from the cabin air on a continuous, non-cyclic basis.
- 2. The output CO2 is free of diluent gas contamination (N2).
- 3. If required, the system is capable of concentrating carbon dioxide from air at any partial pressure down to normal atmospheric concentration of 0.03%.

The Carbonation Cell system is composed of a series of three cell stages, each stage transferring a different gas composition. In the first stage air is supplied to the cathode of the cell and carbon dioxide and oxygen are transferred to the anode. The first stage anode gas is transferred to the second stage cathode. Here, due to the high CO₂ partial pressure, the ion species transferred across the cell changes, and a higher CO₂ percentage is obtained from the second stage anode gas. This mixture is fed to a third stage which preferentially transfers oxygen, leaving essentially pure carbon dioxide.

When completed, two full scale cells for each stage will be mounted in modules which may be individually operated and monitored in a large test stand for short term parametric tests. These tests will be followed by a detailed thermal balance study.

The final program objective is a 250-hour life test for two cells of each of the three stages of the concentration system. If this test is successful it will be extended to a 1000-hour test.

2.0 CARBONATION CELL CONCEPT

2.1 General Principles

When various electrodes and electrolytes are combined to form electro-chemical cells, two general classes of cells may result: (I) chemical cells in which the voltage is due to a chemical reaction occurring within the cell (e.g., batteries); and (2) concentration cells, in which the voltage is due to the free energy decrease associated with the transfer of matter from one part of the cell to another. The TRW "Carbonation Cell" is a specific cell which is typical of class (2) electro-chemical cells. The voltage impressed upon the Carbonation Cell results in concentration gradients of carbon dioxide and oxygen gases at the electrodes.

Consider an oxygen-carbon dioxide concentration cell shown schematically below:

$$O_2$$
 (aP O_2), CO_2 (aP CO_2), M CO_3 M , CO_2 (cP CO_2), O_2 (cP O_2) where:

 aP_{O_2} = anode partial pressure of oxygen

 cP_{O_2} = cathode partial pressure of oxygen

M = metallic porous electrode.

For the above cell the reactions are:

Cathode:
$$1/2 O_2 + H_2O + 2e^- \longrightarrow 20H^-$$
 (1)

Catholyte:
$$CO_2 + 20H \longrightarrow H_2O + CO_3^{=}$$
 (2)

Anode:
$$20H = 1/2 O_2 + H_2O + 2e^-$$
 (3)

Anolyte:
$$H_2O + CO_3 = CO_2 + 20H$$
 (4)

Net cell reaction:

$$1/2 O_2 (cP_{O_2}) + CO_2 (cP_{CO_2}) \longrightarrow 1/2 O_2 (aP_{O_2}) + CO_2 (aP_{CO_2})$$
 (5)

The theoretical electrical energy, 2FE, for the spontaneous, isothermal transfer of 1/2 mole of oxygen and one mole of carbon dioxide from the cathode at pressure ${\rm cP}_{\text{O}2}$ and ${\rm cP}_{\text{CO}2}$ to the anode is given by

$$2FE = -RT \ln \frac{(\alpha P_{O_2})^{1/2} (\alpha P_{CO_2})}{(\alpha P_{O_2})^{1/2} (\alpha P_{CO_2})}$$
(6)

Where:

E is the cell reversible potential

F is the Faraday constant.

In the above derivation the bicarbonate transfer mechanism was not considered since it is important only when cPCO2 is high, as in the second stage cell.

2.2 Three Stage CO₂ Concentration System

To separate carbon dioxide from air at one atmosphere and maintain a carbon dioxide concentration of 0.005 atm. according to reaction (5), it is seen from equation (6) that an external source of energy is required, since $(.2) (.005)^2 < (.333) (.666)^2$; i.e., the reaction is not spontaneous. The three stage concentration system uses a series of three electrically powered cell stages, each stage transferring a different gas composition. The electrolyte in the first two stages is potassium carbonate, while sulfuric acid is used in the third stage.

In the first stage, air is supplied to the cathode of the cell and carbon dioxide and oxygen are transferred to the anode. These gases are transferred through the electrolyte solution as carbonate, bicarbonate, and hydroxyl ions. At the anode hydroxyl ions are discharged and oxygen is evolved. This in turn drives the discharge of CO₂ from carbonate ions to replace the diminished hydroxyl ion pool. Figure 2-1 schematically represents this transfer mechanism. The ratio of the various ionic species depends on the carbon dioxide partial pressure at the cathode. The gas composition at the anode, which depends on the ratio of ionic species, also depends on the cathode carbon dioxide partial pressure. This relationship is shown in Figure 2-2, which is based on experimental data. A cathode gas with 0.5 mole percent CO₂ at one atmosphere has a partial pressure of CO₂ equal to 3.8 mm Hg and yields an anode mixture of 57 mole percent CO₂ and 43 mole percent O₂.

This mixture is transferred to the cathode of the second stage cells. Here, due to a higher CO₂ partial pressure, a greater proportion of bicarbonate ions are formed. With the example above, the anode gas cavity of the second stage cells evolves a mixture of 79% CO₂ and 21% O₂. This mixture is fed to a third stage which preferentially transfers oxygen, leaving essentially pure carbon dioxide. The third stage cell is shown schematically in Figure 2-3. A blower is required to provide air to the first stage only, since the cells produce sufficient gas pressure to transfer gas for subsequent processing.

The system summarized above is capable of concentrating carbon dioxide from air at any partial pressure down to the normal atmospheric concentration of 0.03%. At a carbon dioxide partial pressure of 3.8 mm Hg, corresponding to 0.5% CO₂ at one atmosphere, the power required to concentrate carbon dioxide with the three-stage Carbonation Cell is 157 watts/man at a weight of approximately 10 pounds/man.

Figure 2-4 presents a schematic of the three-stage Carbonation Cell carbon dioxide concentration system.

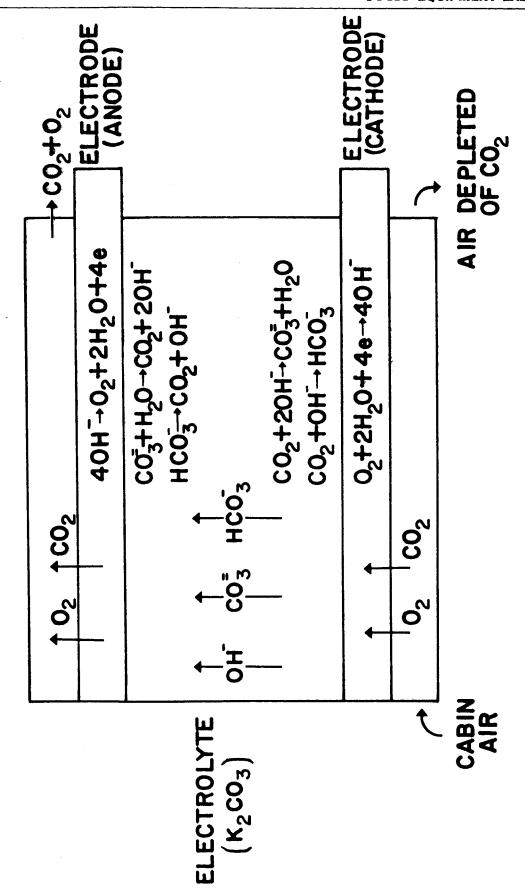


FIGURE 2-1 STAGE I CELL - SCHEMATIC REPRESENTATION

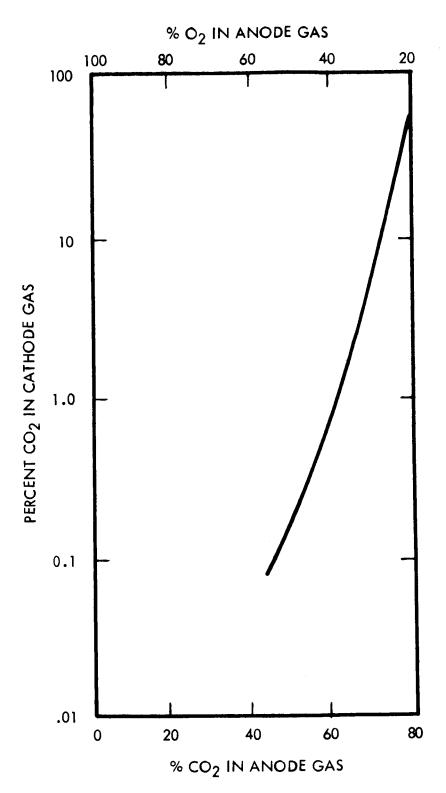


FIGURE 2-2 ANODE GAS MIXTURE AS FUNCTION OF CATHODE GAS MIXTURE FOR CARBONATION CELL

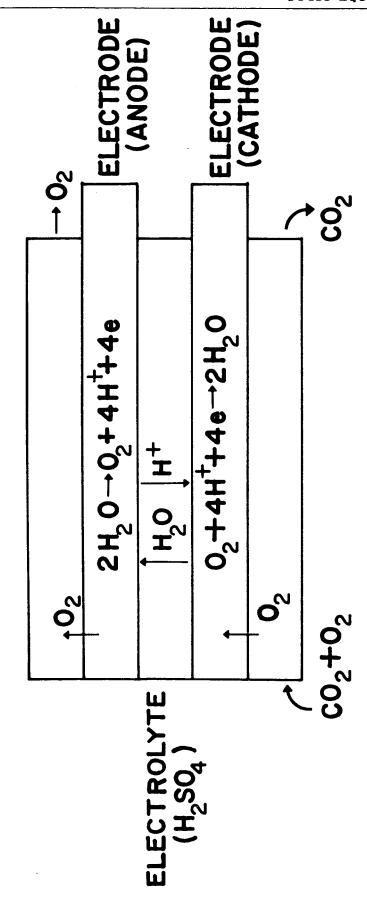


FIGURE 2-3 STAGE III CELL - SCHEMATIC REPRESENTATION

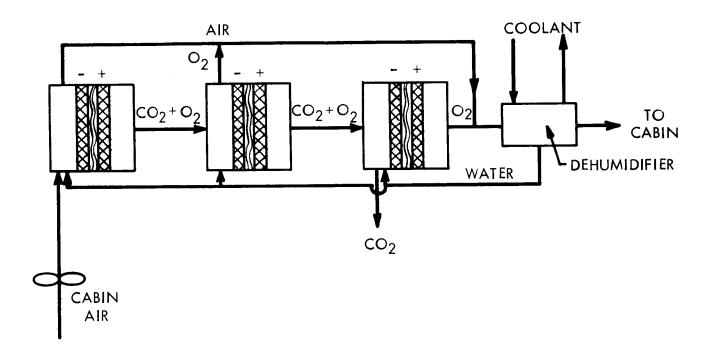


FIGURE 2-4 SCHEMATIC OF CARBONATION CELL CARBON DIOXIDE CONCENTRATION SYSTEM

3.0 DESIGN DISCUSSION

3.1 Preliminary Analysis of Four-Man Prototype

To determine the basic characteristics of the large experimental cells, an analysis of a four-man prototype system was performed. The major objective of this preliminary analysis was to evolve a control method which would allow for both the thermal and humidity balance to be accomplished within the cell. A further consideration was to exercise this control without the aid of external cooling or humidification, thereby minimizing system complexity and weight.

A schematic of the design model used in this analysis is shown in Figure 3-1. Since the process gas entering the cathode chamber is at a lower dew point temperature than the cell electrolyte, water is evaporated from the membrane surface to the gas stream. Thus the heat of vaporization of water is used to remove the heat generated within the cell. The equilibrium partial pressure of water vapor that obtains in the gas cavity will determine the mean cell electrolyte concentration and temperature.

The equilibrium partial pressure of water vapor will be a function of the process gas flow rate, dew point, and the cell terminal voltage. Analysis indicates that the above method of control is applicable to the second and third stage units but is a marginal control scheme when applied to the first stage unit. For example, a 1st stage unit operating at relatively high process gas flow rates (5 x stoichiometric CO₂) and low dew point temperatures (30% R. H.) would approach an equilibrium temperature near or below ambient and electrolyte concentrations not far removed from saturation.

An alternate approach for the thermal control of the 1st stage unit would be in the use of the process gas to remove cell heat. The humidity control would have to be accomplished by external means. In this mode of operation the process gas will experience a temperature increase at a constant dew point temperature which in turn will cause a gradient in the electrolyte concentration. The magnitude of this gradient is a strong function of the process gas flow rate. Calculations indicate that allowable concentration gradients are realized only at flow rates approaching 10 x stoichiometric CO₂.

The preliminary analysis summary presented in Table 3–1 was based upon several simplifying assumptions:

- a) No external heat transfer to or from the cell.
- b) Linear thermal and concentration gradients within the cell, if any.
- c) 1st stage ion transfer 100% CO_3^- ; 2nd stage ion transfer 100% HCO_3^- .
- d) Negligible heat transfer from cell to process gas in 2nd & 3rd stage.
- e) Instantaneous mixing of process gas and of evaporation water in 2nd and 3rd stage.

TABLE 3-1

PRELIMINARY ANALYSIS SUMMARY

	Cabin Air		Stage 1			Stage			Stage 11	
	to Pre-	පී	Cathode	Anode	ပြ	Cathode	Anode		Cathode	Anode
	conditioner	드	Out	0 ∩ †	드	Out	Οnŧ	드	Out	Out
Design Conditions for 4-Man System										
Carbon Dioxide ∼ gm moles∕hr	40	40	36	4	4	0	4	4	4	0
Oxygen ~ gm moles/hr	1680	1680	1678	2	2	_	_	-	0	
Nitrogen ~ gm moles/hr	3700	3700	3700	,	ı	ı	•	•		ı
por	85	935	934	_	-	2.4	11.9	11.9	20.05	5.1
Gas Temperature ∼° C	20	46	51.7	58.8	58.8 82.5	82.5	82.5	82.5	87.4	87.4
Gas Dew Point ∼°C	8	46	46	46	46	46 80.5	80.5	80.5 85.0	85.0	85.0
Cell Temp. ∼•C		99	2.19		82.5 82.5	82.5		87.4	87.4	
Electrolyte Concentration ~ wgt%		45.3	52.5		17.2	17.2		20.0	20.0	
Power Density ~ watts/ft2		20			50			20		
Total Area \sim ft ²		6			3			3		
Cell Power ∼ watts		300			150			150		
Design Conditions for Prototype Cells										
Cell Area \sim ft 2					, -			_		
Maximum Current Density ~ ASF		20			20			20		
Maximum Gas Flow \sim cfm/cell		8.5			0.5			0.5		
Cooling Method		Process Gas	s Gas		Water Evap.	Evap.		Water Evap.	Evap.	
		and			and			and		
		Conduction	ction		Conduction	ction		Conduction	ction	

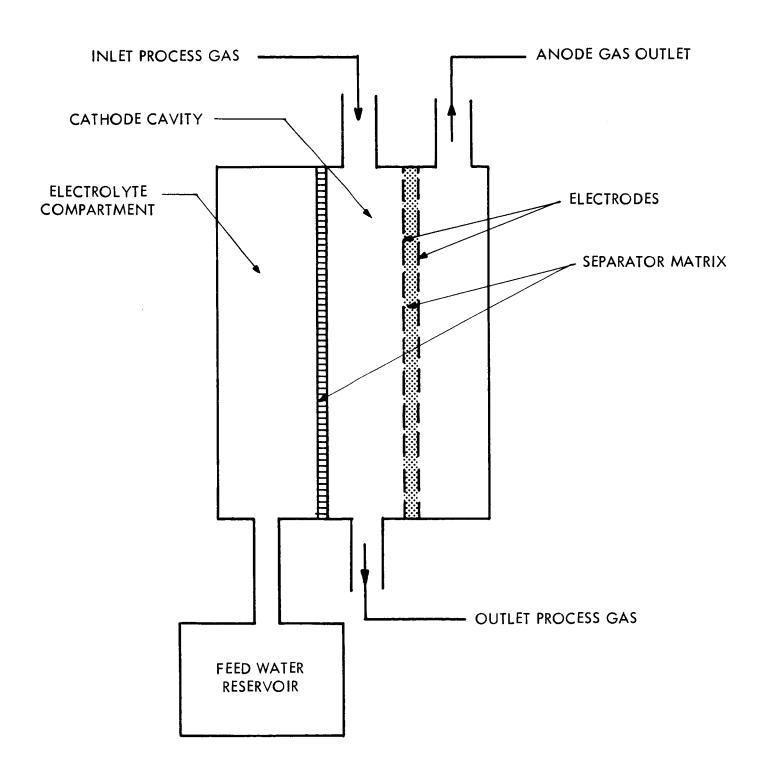


FIGURE 3-1 CELL DESIGN MODEL FOR PRELIMINARY ANALYSIS

A complete thermal and humidity analysis will be performed in Task IV based on the results of the experimental studies to examine all three stages operating with variable parameters.

3.2 Experimental Test Cells

For use in the experimental program, 12" x 12" active area cells were designed. These cells use American Cyanimide fuel cell electrodes measuring 12" x 12". (Types AA1 and AB6X are being evaluated.) The logic behind using large cells is to prevent any scale-up problems in going from experimental cells to a flight configuration. Consistent with the desire to build experimental cells which correspond with flight cells, materials have also been selected with this in mind. The cells for Stage I and Stage II will therefore use gold plated magnesium to yield a low weight system. The Stage III acid cell materials problems are discussed in Section 4.0.

Although experimental cells will all be single cell units, all porting and flow patterns have been designed so that bipolar plate designs could be substantially copied from the experimental cell designs. Experimental cell components have also been designed to reduce the number of drawings required and to permit interchangeability of components as much as possible. To accomplish this, three basic end plate designs were necessary an anode plate for use on all three stages; a first stage cathode plate to accommodate the high flow of the first stage; and a cathode plate for the Stage II and IIIcells which incorporates a wicking system for water addition and is designed for lower gas flow than the first stage cathode.

The cell end plate, fabricated of a single piece of magnesium, combines a number of functions into the single piece:

- a. Cell end sealing plate
- b. Pressure clamping plate
- c. Gas distribution (manifolding and pin structure)
- d. Current collector
- e. Integral structure for attachment of flow line fittings.

Uniform compression of the cell matrix and good electrical contact with the electrode is provided by the pin structure (0.06" square pins). The cells are sealed with a large diameter O-ring and assembled with 28 bolts to assure uniform compression.

No baffling for flow pattern control is incorporated in the cells. Baffling will be added during assembly by inserting Teflon tubing between adjacent rows of pins thereby providing a ready means for evaluating various flow patterns.

Upon referring to Figures 3-2 and 3-3, Cell Assembly Drawings, the configuration of the cell may be seen. The active portion of the cell, composed of two electrodes (one on each side of an asbestos electrolyte matrix), is compressed between two cell end plates. The current leads are attached directly to the end plates, with electrical contact to the electrodes being through the pin structure. Electrically insulating washers are used on the compression bolts to insulate one end plate from the other. The Teflon tape shown on the assembly drawings is used to establish the desired end plate spacing and electrolyte matrix compression.

Cooling of the Stage II and IIIceII will be by water evaporation which is provided for by an asbestos wick used to separate a water cavity from the cathode gas compartment. Evaporation is off the face of the wick. The wick is held firmly in place by tantalum screening on either side of the asbestos. The asbestos-tantalum screen wick assembly is clamped between the pins of the end plate water cavity and the pins of the gas diffusion plate. The gas diffusion plate, shown in Figure 3-4, provides an even flow distribution of cathode gases between the wick and electrode surfaces. Electrical continuity to the electrode is from the end plate to the rim of the gas diffusion plate and then to the pins on the diffusion plate touching the electrode.

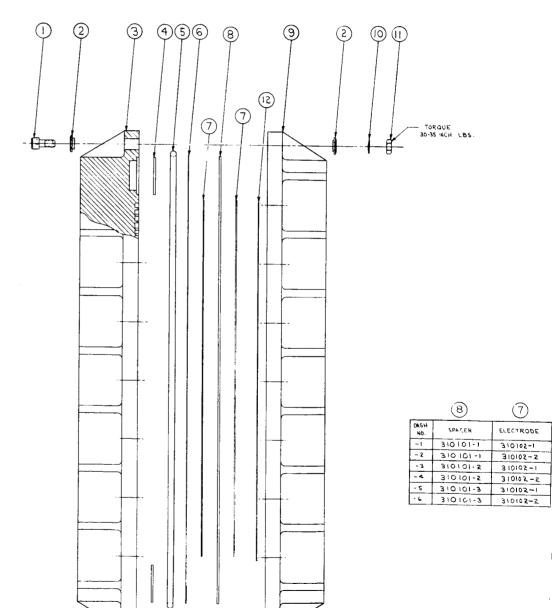
The gas distribution system has been designed so that each cell can handle five times stoichiometric flow at 50 amperes per square foot with a maximum pressure drop of 0.2 psi. To control the flow pattern the major drop is across the distribution holes connecting the flow channels with the cell field. The field drop is very low and therefore non controlling. Drops of water or irregularities in the cell will therefore not materially affect the flow patterns. Inlet ports and manifolding on the cell end plates are seen on the photographs of the representative experimental cell components, Figures 3-5, 3-6, and 3-7.

The small recession provided in each manifold slot is for insertion of a plastic seal plate to complete each manifold chamber. Clearly shown on the back of the end plates are provisions for:

- a. Attachment of power lugs
- b. Mounting of thermocouples
- c. Connection of pressure gauges
- d. Attachment of inlet outlet flow line fittings
- e. Installation of liquid level sight glass for the Stage II & III water cavity.

The large two-inch diameter holes on the back side of the plates are merely holes to decrease the weight of the structures. Design of the holes was such as to minimize machining requirements and to insure that cell deflection at center of cell would not exceed 0.001 inch when all 28 bolts are properly tightened. This lightening structure is by no means optimum. For a flight unit a much more intricate cast type pressure clamping plate would be used.

13



ELECTRODE 310102-1

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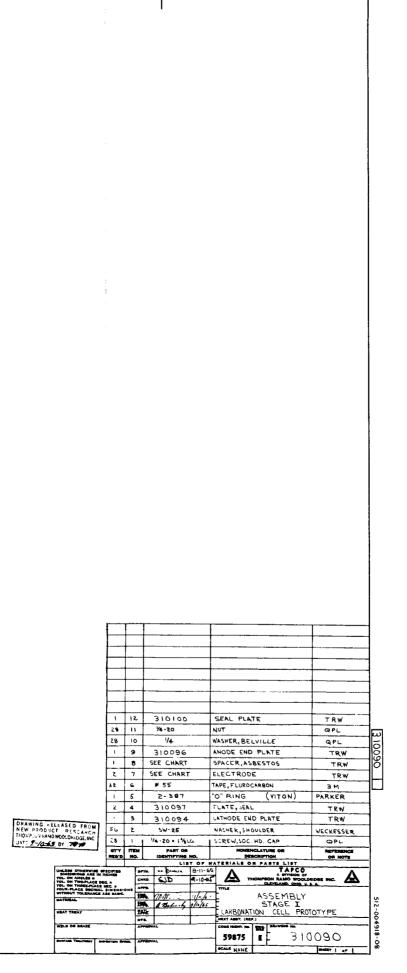
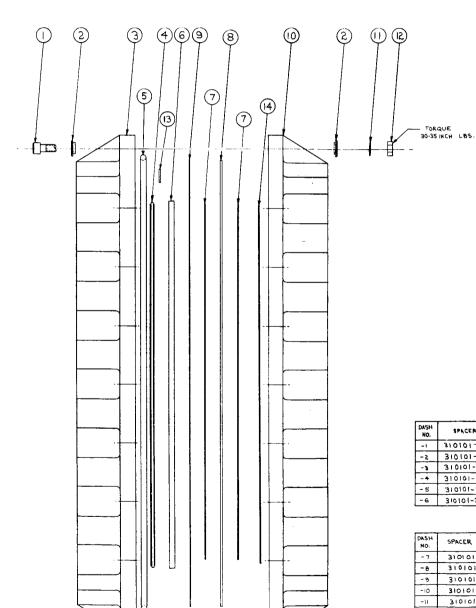


FIGURE 3-2



STAGE II

		STAGE II
DASH No.	SPACER B	ELECTRODE()
-1	310101-1	1-501018
-5	310101-1	310105-5
-3	3-10101-2	310105 -1
-4	3-10101-5	3-201016
15	310101-3	310102-1
-6	310101-3	3-501018

STAGE III

		SIAGE III
ASH NO.	SPACER 8	ELECTRODE 7
-7	310101-1	310105-1
- e	310101-1	310102 -2
- 95	310101-2	310102-1
-10	310101-2	310105-5
-11	310101-3	310105-1
-12	310101-3	310102-2

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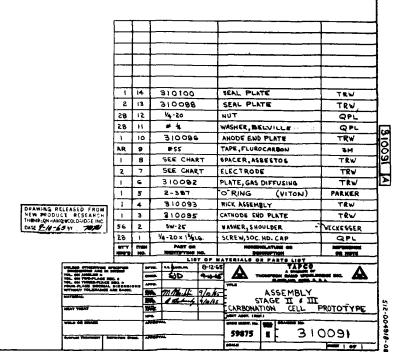
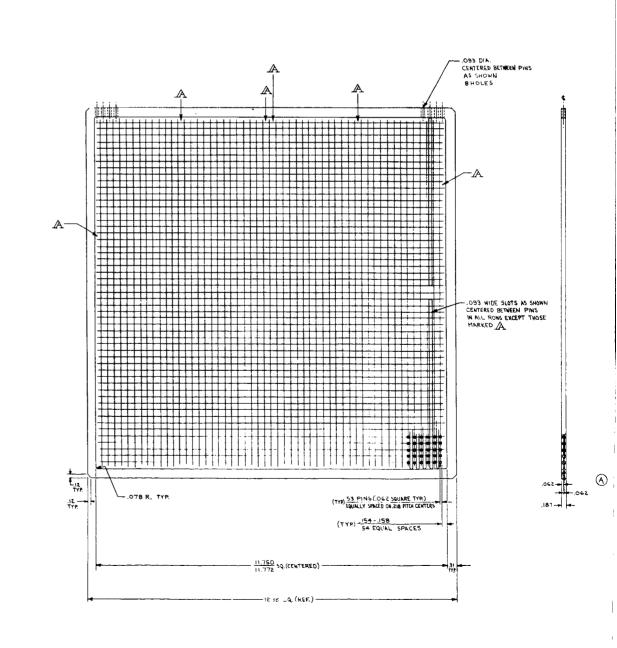
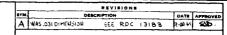
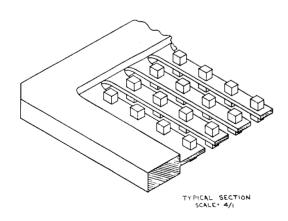


FIGURE 3-3



TRW EQUIPMENT LABORATORIES





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FIGURE 3-4

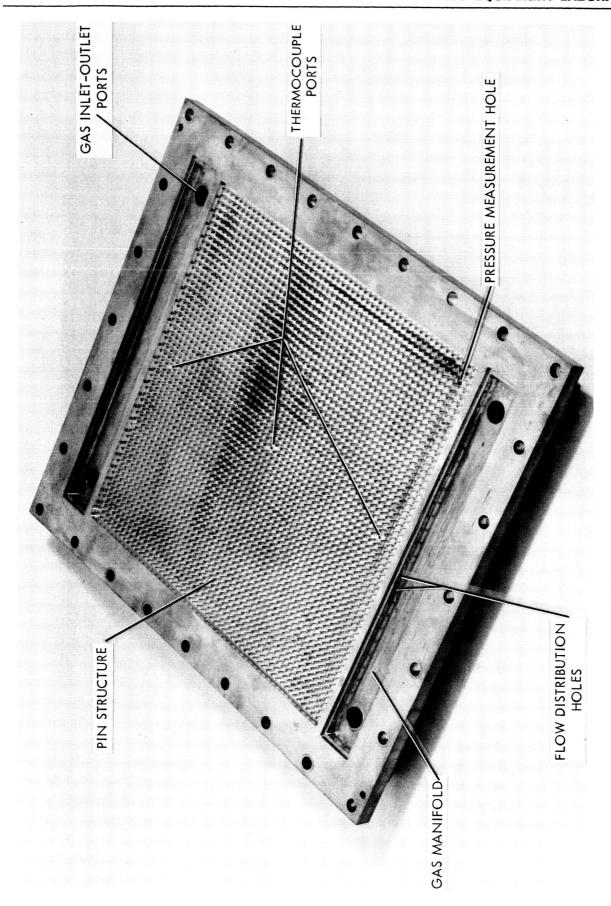


FIGURE 3-5 STAGE I CELL CATHODE END PLATE

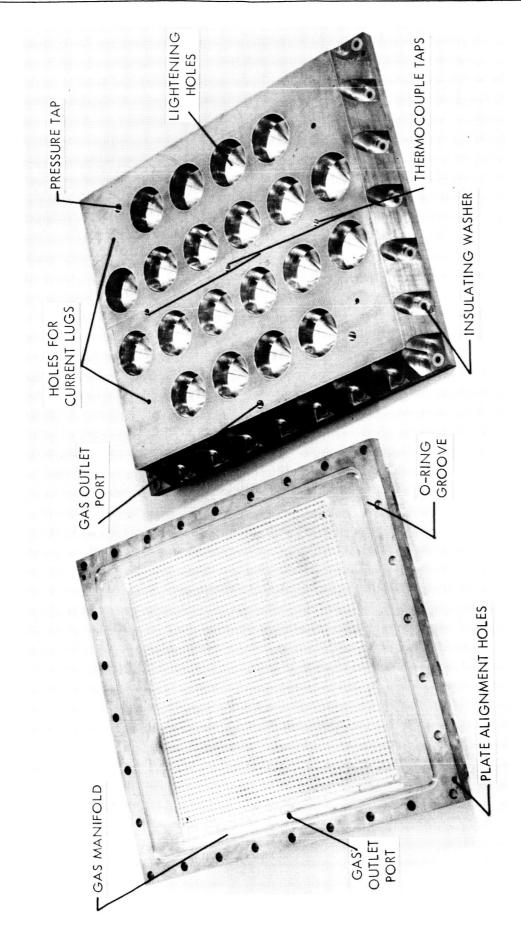


FIGURE 3-6 STAGE I, II, AND III CELL ANODE PLATE

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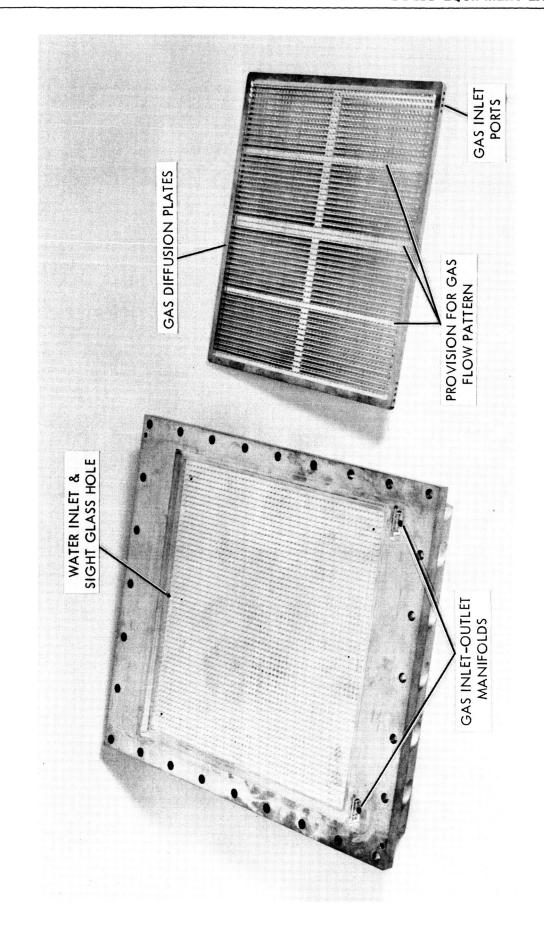


FIGURE 3-7 STAGE II AND III CELL CATHODE AND GAS DIFFUSION PLATE

3.3 Test Stands

- 3.3.1 Small Cell Test Rig Small test cells are used to check out problem areas expected on the large experimental cells and to examine in detail unexpected or strange performance in the large cells. Figure 3-8 presents a schematic of the set-up used for this testing. Provisions are made for precise control of cell temperature and feed gas humidity. Inlet gas composition is known (analyzed cylinger gas) and the outlet gas composition is monitored by an oxygen analyzer. Provision is made for inlet and outlet flow rate measurement. Figure 3-9 presents a schematic of the electrical measuring circuits used to analyze cell electrical performance. Figure 3-10 presents a photographic view showing the major components of the test rig.
- 3.3.2 Large Cell Test Stand The large cell test stand was designed such that cells could be tested individually during the parametric short term testing and also used for life testing of six cells concurrently. Figure 3-11 presents a schematic of the full test stand. As indicated, gas feed to the first stage will be by a mixture of laboratory air and bottled CO₂. Stage II and III gas feed is from premixed bottles. A large vacuum pump is connected to a common manifold connecting the gas outlets of all three stages to maintain cell operating pressures in the necessary sub-ambient range. Common manifolding enables the use of a single O₂ analyzer, CO₂ detector, and gas chromatograph to measure the composition of gases in each stage. Individual humidity detectors are provided for each stage.

Figures 3-12 and 3-13 are photographs of the test stand. One end of the stand, Figure 3-12, is the control panel, which will be used to set gas flow rates, control temperatures, and route gases to gas analysis instrumentation. In Figure 3-13 is seen the main portion of the stand. The bottom houses the plumbing, vacuum pump, gas humidifiers, and cell power supply. The upper portion consists of three lucite-enclosed sections which will house the test cells. Each section will enclose two cells of one stage.

Individual cells will be installed on modules for ease in installation in the test stand. Each module will consist of two cells, a small frame, instrumentation, and all necessary fluid and electrical connections. The modules will then be installed in the test enclosure by merely connecting four tube fittings, electrical interconnections, and thermocouple jacks. Figure 3–14 is a photograph of a partially assembled module.

3.3.2.1 Thermal Control - An independent ambient air temperature control, which has a range up to 175° F, is provided for each stage. Heat is supplied in the top of each enclosure by nine light bulbs (300 watts per bulb). Seven of the bulbs will have their level controlled by a variac while the remaining two will be cycled on-off by a temperature controller to maintain desired enclosure temperature level. The air heated by the bulbs is circulated by two externally mounted blowers per stage. The hollow framework members of the test stand form the duct work which carries the hot air from the top of the enclosure to the bottom of the enclosure and onto the test cells.

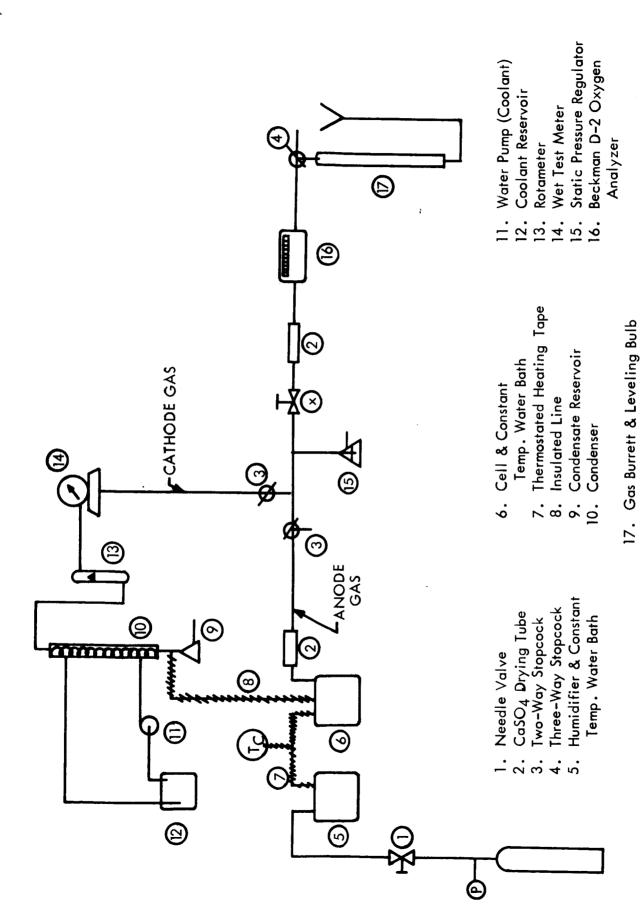
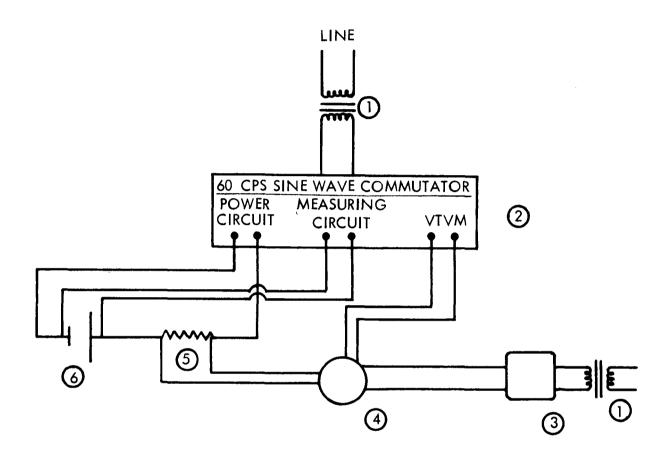
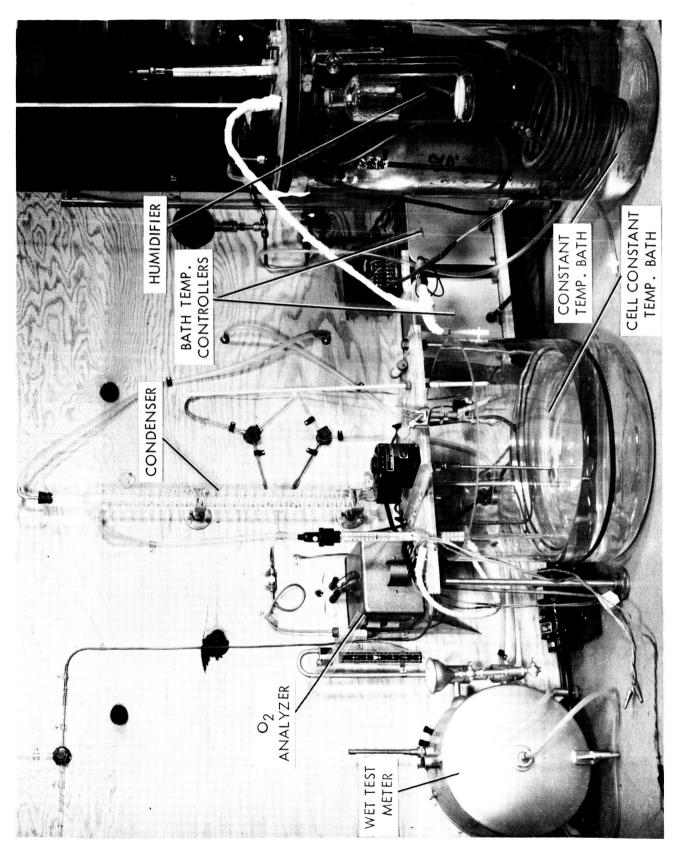


FIGURE 3-8 TEST STAND SCHEMATIC - SMALL EXPERIMENTAL CELLS

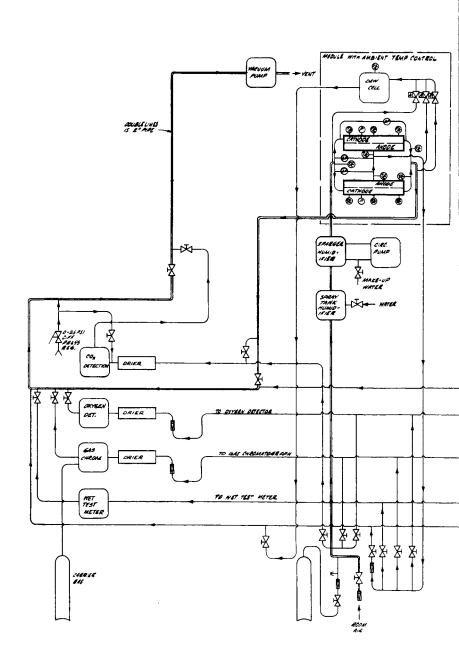


- 1 CONSTANT VOLTAGE TRANSFORMER
- 2 TRANSISTORIZED 60 CPS SINE
 WAVE COMMUTATOR. (POLLNOW, KAY, JOUR. ELEC. SOC.,
 109, 648)
- 3 JOHN FLUKE DIFFERENTIAL VOLTMETER
- 4 ROTARY SWITCH
- 5 WESTON 50 MV 5 AMP PRECISION SHUNT
- 6 TEST CELL

FIGURE 3-9 ELECTRICAL TEST MEASURING CIRCUIT



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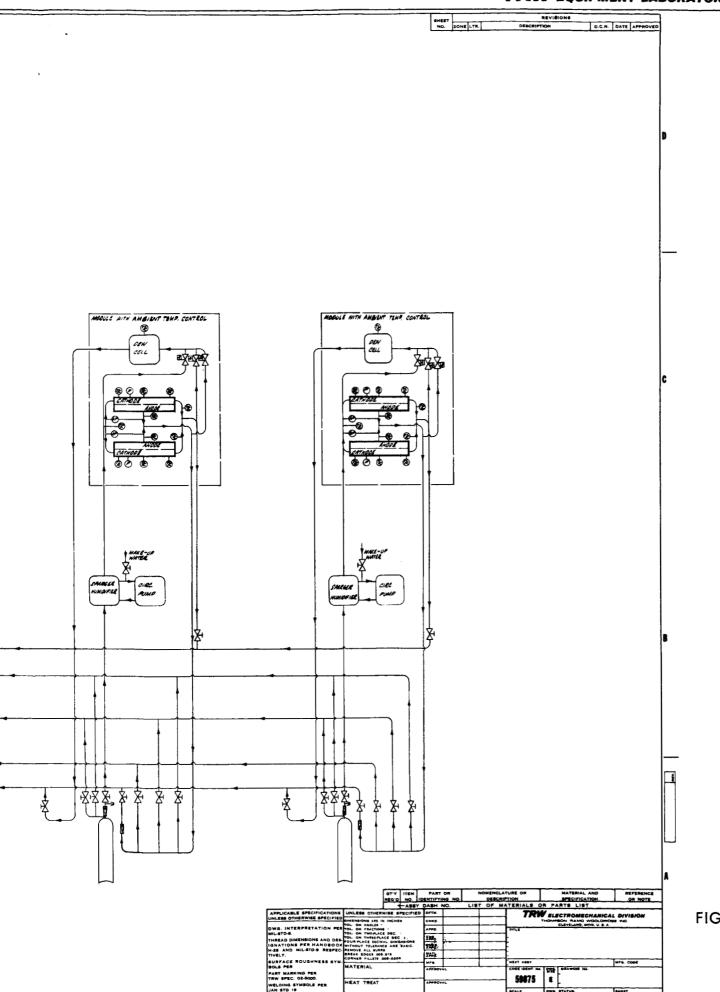


FIGURE 3-11

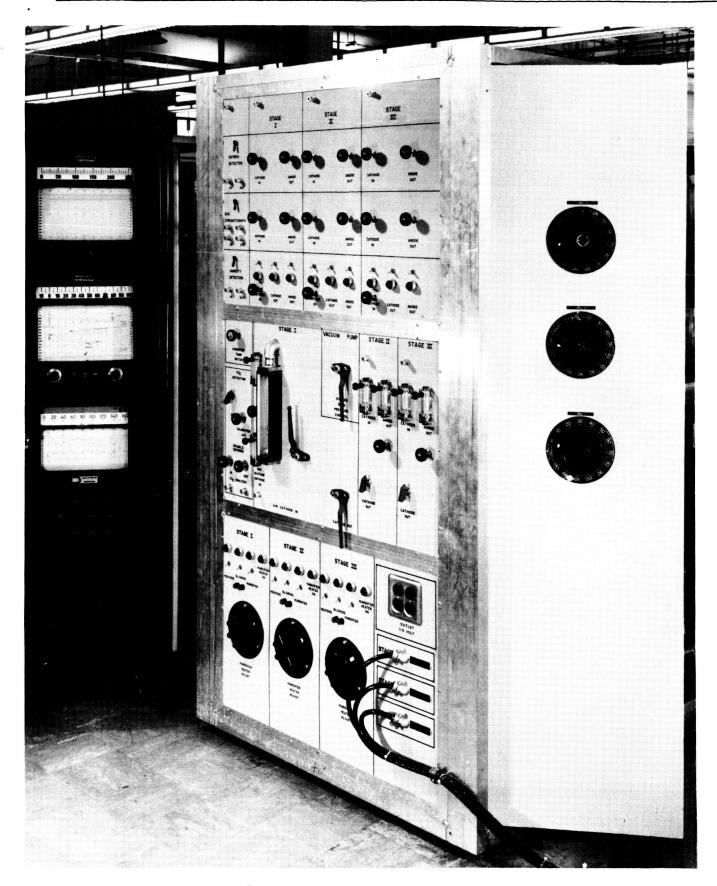


FIGURE 3-12 TEST STAND CONTROL PANEL

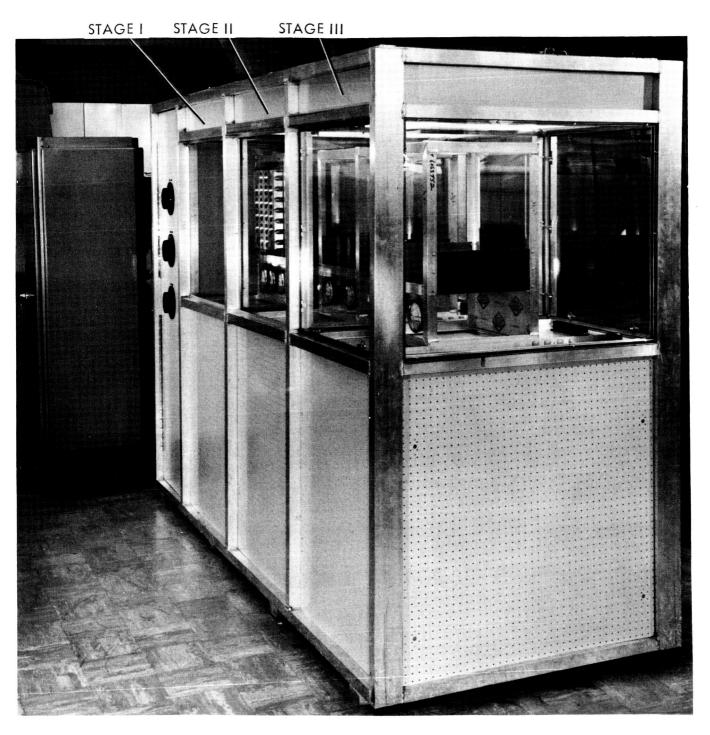


FIGURE 3-13 TEST STAND

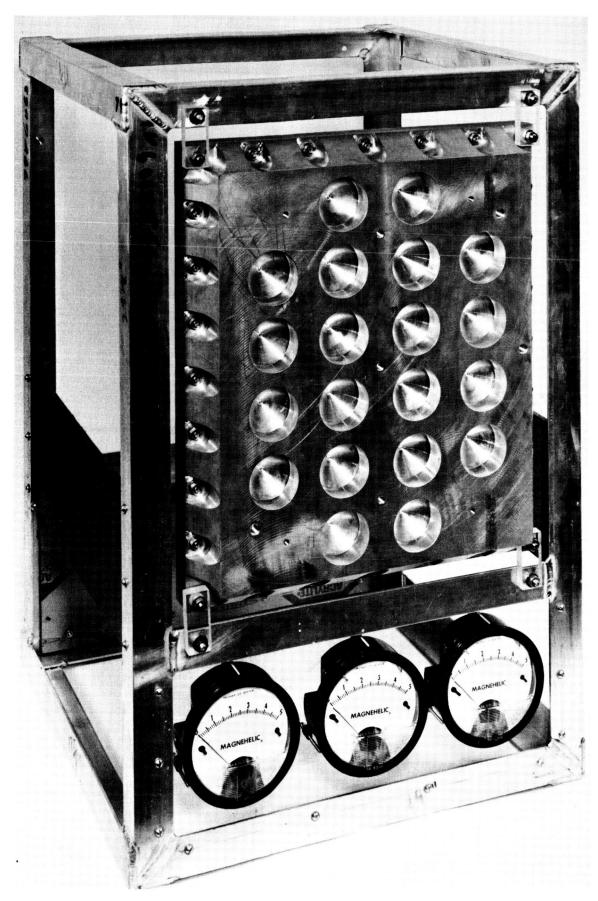


FIGURE 3-14 STAGE I MODULE - ONE CELL MOUNTED

3.3.2.2 Humidity Control - Precise control of inlet gas humidity is necessary in order to study cell moisture balance. It is accomplished by passing inlet gases through thermally-controlled water baths, measuring 30" tall and 10" in diameter. The gas passes through spargers in the bottom of each tank. The gas passes upward through the bath which is maintained at a controlled uniform temperature by a temperature controller, variac, heater, and mixing pump. At the exit a baffle arrangement stops droplet carryover.

The large gas flow of the first stage necessitates the use of a spray tank to aid in prehumidification. As shown in Figure 3-11, gas in the first stage passes through the spray tank before it enters the controlled humidification unit.

Figure 3-15 is a photograph of the test stand with the bottom panels removed to expose some of the components used to control temperature and humidity.

3.3.2.3 Instrumentation -

a) Gas Analysis

A "Critical Orifice Carbon Dioxide Analyzer" (Harvard Apparatus Co., Dover, Mass.) will be used to monitor the carbon dioxide content of the inlet and outlet 1st stage process gas. The instrument has a range of 0-10% CO₂ with a stated accuracy of \pm 0.05% CO₂. The unit can be adapted so as to read the CO₂ content on a continuous basis.

The "Beckman E-2 Oxygen Analyzer" will also be used. It is a continuous sampling multirange instrument which determines oxygen partial pressure by measuring the sample's magnetic susceptibility. This unit, together with suitable manifolding, will monitor the anode gas streams of all stages. The carbon dioxide content of the gas stream can then be determined by difference (in the absence of internal gas leakage).

A "Beckman GC-2A Gas Chromatograph" will be primarily used as a cell leak detector for the 1st stage unit (detect the presence of N_2 in the anode stream). The unit will also periodically sample all gas streams to establish purity levels.

- b) Dew Point Temperatures. The dew point temperature of any one of the three gas streams associated with a given stage will be continuously monitored by a "Honeywell dew probe sensor" (SSP129-C). The sensor is rated at a maximum dew point temperature of 160°F.
- c) Cell Temperature Voltage and Current will be continuously recorded with a multipoint potentiometric recorder. The use of suitable voltage divider circuits and precision shunts will determine the operation range of the various channels.

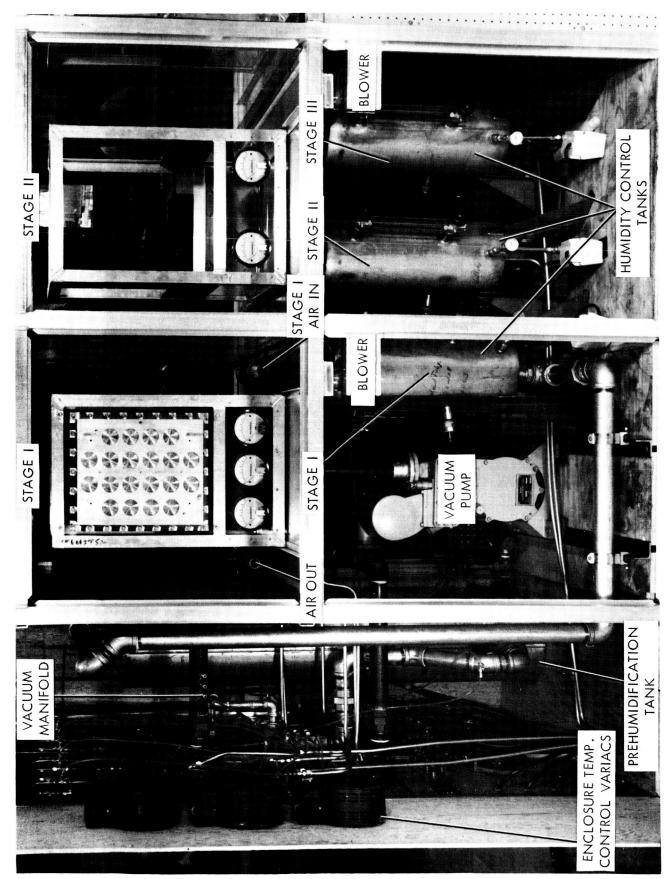


FIGURE 3-15 THERMAL AND HUMIDITY CONTROL COMPONENTS

- d) Gas Flow Measurement. All cathode flows will be monitored by rotameters of the variable area type. Anode gas flows will be monitored with a precision scientific wet test meter.
- e) Pressures. Cell operating pressures will be measured by compound type pressure gauges while differential pressure measurements will be made by "Dwyer Magnehelic Gauges". These gauges will be mounted on the individual modules.

4.0 MATERIALS

4.1 Stage I and II Cells

Based on experience at TRW on similar cells, material selection for the Stage I and II cells was quite straightforward. A compilation of these materials is given in Table 4-1.

TABLE 4-1

STAGE I AND II CELL MATERIALS

Component	Material	Remarks
End Plates	AZ31B Type 2 Magnesium Plate	Gold Plates as follows a) Zinc immersion (molecular) b) 0.1 to 0.3 mil copper c) 1.5 mil nickel d) 0.03-0.05 mil electroless gold.
Electrodes	American Cyanamid Type - AB6	Gold plated nickel screen with 9 mgm/cm ² of platinum
Electrolyte <i>M</i> atrix	Matted Structure	Whatman GFB glass fiber Filter Paper – (20 mils thick)
O-Ring	Neoprene	
Gas Diffusion Plate	Same as End Plates	
Wick Assembly	Tantalum Wire screen and glass fiber paper	
Seal Plates	Epoxy Fiber Board	

4.2 Stage III Acid Cell

The Stage III material selection is a difficult problem in view of the sulfuric acid electrolyte and high cell operating potential (anode).

4.2.1 <u>Materials Screening</u> – A material screening and evaluation program was conducted in an effort to select a cell material for the Stage III cell construction. The material selected should meet the following requirements:

- a. Resistant to sulfuric acid corrosion under electrochemical action.
- b. Low cost raw material
- c. Easily machinable into intricate forms
- d. Sufficient mechanical strength
- e. Electrical conductor
- f. Impervious to gas
- g. Available in proper form and within schedule framework.

Table 4-2 lists the materials which were considered for possible use on the program. They were all eliminated from consideration for not meeting one or more of the above listed requirements. This then forces the use of end plate assemblies which are composed of a number of different pieces fabricated of different materials. Table 4-3 gives the materials to be used for Stage III cell end plates. Some minor design modifications must be made to accommodate the use of these materials.

TABLE 4-3

STAGE III CELL END PLATE MATERIALS

<u>Part</u>	<u>Material</u>	Remarks
Pin Structure	Polyvinyldichloride (PVDC)	Thick enough to accept all fittings
Pressure Clamp- ing Plate	Magnesium Plate	Unplated
Current Collector	Gold Plated Copper Grid	Embedded in pin structure electroplated gold 2 mils thick

4.2.2 Non Porous Gold Plated Magnesium Evaluation - Except for the high cost of gold plating, magnesium with a 2-mil thick non-porous gold plating would be our choice for the third stage end plates. As such, test samples have been put through a number of corrosion tests.

TABLE 4-2

STAGE III CELL-MATERIALS EVALUATION

	0	Cost	Avail	Availability	Mech.	Elec.	
Structure Type	Material	Fabrication	Size	Schedule	Prop.	Prop.	Remarks
Carbon – ATJ Type	Low	Low	Yes	Yes	* ° Z	Poor	Gas permeability too high, weak and brittle
Carbon – High Density Type (ER–1924)	High*	Low	* Ž	<u>°</u>	Ques- tionable	Good	Cost too high – not available in large enough pieces
Gold Plated Carbon	Moderate	Low	\ \ \ \	Yes	Ques- tionable*	Good	Questionable Strength
Magnesium – Non Porous Gold Plate	High*	Low	Yes	Yes	Good	Good	Gold plating cost too high – would be used for flight hardware.
Electroclad Tantalum on Carbon Pin Structure	Moderate	Moderate	Yes	Yes	Good	Ques. *	Use of Tantalum Questionable – See Section 4.2.3.
Other Inert Metals	High*	Moderate	Yes	Yes	Good	Good	Material cost too high
Plastic Pin Structure – Gold Plated Current Collector	Low	Low	Yes	Yes	Satis- factory	Good	Must use multiple type structure – will be used for experimental cells
*Major reasons for elimination	nation						

Three $3" \times 5"$ magnesium test plates containing a machined pin structure were plated as follows:

- a. Zinc immersion (molecular)
- b. 0.1 to 0.3 mil copper
- c. 1.5 mil nickel
- d. 0.5 mil gold

All of the above plating was done by Furniture City Plating Co. except for the gold plating (SEL-REX-CO.) The gold plate was applied by electrolytic deposition. The results of several corrosion tests are summarized in Table 4-4. All tests involved immersion of one half of the test plate in a 6 normal aqueous solution of sulfuric acid. The test plate served as the anode of an electrolysis cell couple. Figure 4-1 is a photograph of the test samples as described below.

A post test (no. 1) microscopic examination of the unused portion of plate #1 revealed several pin holes which may have been the centers of attack. The brownish deposit observed in tests no. 1 and no. 4 is an oxide of gold which readily formed at the operating potential of 1.8 to 1.9 volts with respect to a standard calomel electrode. This brown oxide is easily rubbed off leaving a smooth unpitted surface (Figure 4-1, plate no. 2). The pinkish coloration imparted to the test solution is probably due to the formation of a gold sol, formed by the reduction of gold oxide particles which migrate to the cathode by forced convection.

The results of tests 2 to 5 indicate that the base metal is well protected from either chemical or electrochemical attack. In test no. 5 a platinum electrode was placed in intimate contact with the test plate simulating a typical cell assembly. No formation of gold oxide was evident, indicating that the test plate is functioning only as an electron exchange medium and is not involved in any electrochemical reaction. The small black spots on the test sample used in test no. 5 are the points at which the electrode was spot-welded to the gold plated magnesium.

Further evaluation of the gold plated magnesium will be conducted by the use of a small test cell fabricated of this material.

- 4.2.3 Small Cell Tests Small cell testing was to be conducted to examine those areas which are problems in the large cell testing program. The small cell testing program is also intended to examine in detail large cell test results which seem to be at variance with expected results. During this first phase of the present program only the materials problem of the third stage cell has been studied by testing of two cell types:
 - a. Carbon end plates
 - b. Plastic end plates with tantalum current collector inserts

TABLE 4-4

CORROSION TESTS GOLD-PLATED MAGNESIUM

Test No.	Temp.	Test Duration (Hours)	Plate Potential (Volts)	End of Test Observations Solution	vations Test Plate	Solution Analysis
1 Plate #1	25	15	+1.9 at 0.5 amps (constant)	Pinkish coloration brownish – purple particles dispersed in solution	Brown adherent deposit, random distribution of pin holes. Severe attack at one point on edge of plate.	High Ni, Mg, Ca, Cu, Fe, Au.
2 Plate #2	52	72	None	No change	No change	ļ
3 Plate #2	06	29	None	No change	No change	
4 Plate #2	25	21	+1.8 at 0.5 amps (constant)	Pinkish coloration	Brown adherent deposit. No pin holes observed.	
5 Upper half Plate #2 AA-1 Elec. Spotwelded to Plate	25	23	+1.8 at 0.5 amps (constant)	No change	No deposit on plates. No pinholes observed. Black spots due to spotwelding electrode in place.	None

(1) With respect to a saturated calomel electrode, counter electrode - 50 mesh tantalum screen. Potential given is minimum at start of test — a slight increase with time occurred but was not measured.

(2) Spectrophotometric

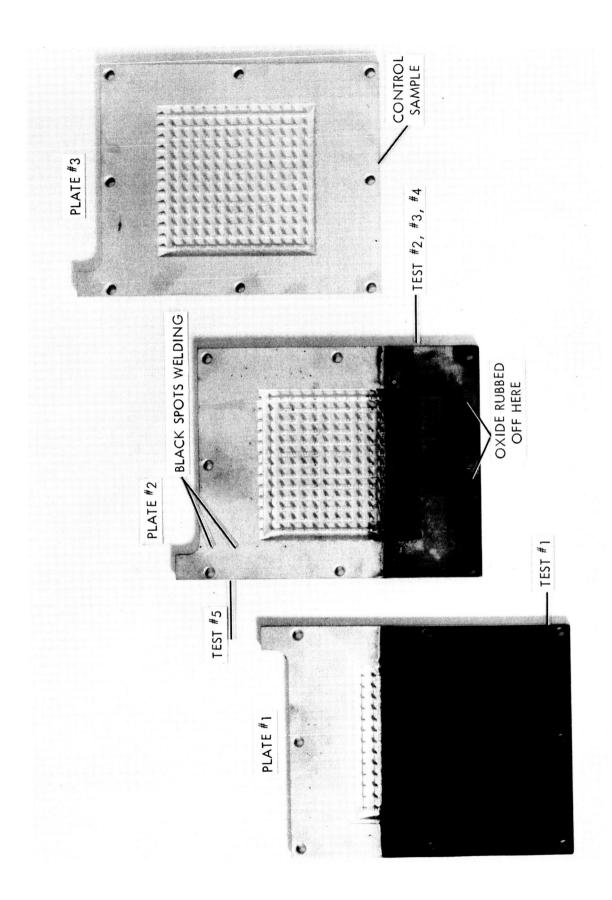


FIGURE 4-1 GOLD PLATE EVALUATION TEST SAMPLES

- 4.2.3.1 Carbon Cell A set of carbon end plates was fabricated to study the applicability of using readily available grades (ATJ) of carbon for the Stage III end plates. No electrical performance tests were conducted as the permeability of the carbon end plates allowed leakage of gases at operating pressures. The low mechanical strength of the carbon is evident in Figure 4–2, as the upper end plate is cracked where the current lug had been attached to the back of the cell. Further tests with carbon are not contemplated.
- 4.2.3.2 Plastic Cell with Metal Inserts A total of 35 hours of testing has been accumulated on the plastic test cell (Figure 4-2). Lucite end plates containing an integral pin and baffle structure). The current collector recessed in the end plate consists of a vertically ribbed tantalum frame. All testing was conducted in the test system described in Section 3.3.1. Test conditions were as follows:

Electrodes: AC #AA-1 (Active area 127 CM²)

Electrolyte: 6N H₂ SO₄/Whatman #GF-B Glass Filter Paper

Cell Pressure: Ambient

Cell Temperature: 35° C ± 0.1° C

Current Density: 31.5 MA/CM²

Cathode Gas: 80% CO_2 - 20% O_2 - 276 cc STP/min - 4 \times stoichiometric (O_2)

The best performance obtained was 1.0 volts terminal after 8 hrs. operation at 31.5 MA/CM². In all tests a polarization-time dependence was noted of the order of 0.5 MV/min. Figure 4-3 is a plot of the cell "IR" free voltage as a function of time at load for a typical run.

Difficulties were encountered in sealing the test cell (internal). As a result, an accurate determination of the anode gas purity is still to be made. Additional difficulty was encountered in maintaining proper cell dew point owing to the fact that the plastic cell acts as an insulator and prevents the internal temperature of the cell from matching the water bath temperature.

Several possibilities exist as to the cause of the polarization-time dependence:

- a. Progressive oxidation of the tantalum screen and current collector, resulting in an increased overall cell resistance.
- b. Reduction of the carbon dioxide at the cathode, with the resulting formation of carbon monoxide the carbon monoxide being irreversibly adsorbed on the electrode.
- c. Internal cell temperatures higher than the ambient would result in a recession of the electrode-electrolyte interface since the entering gas dew point is based upon ambient cell temperature.

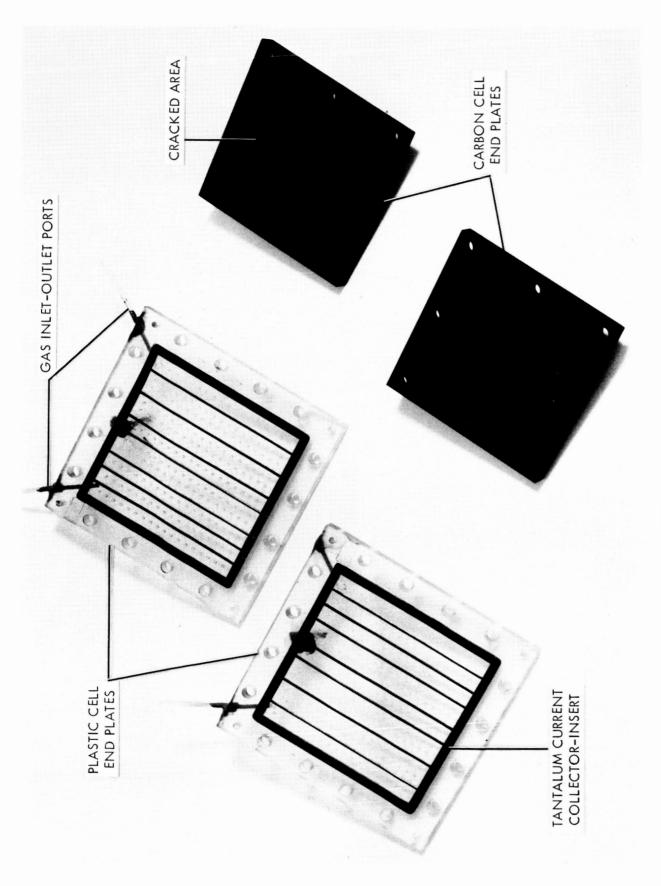
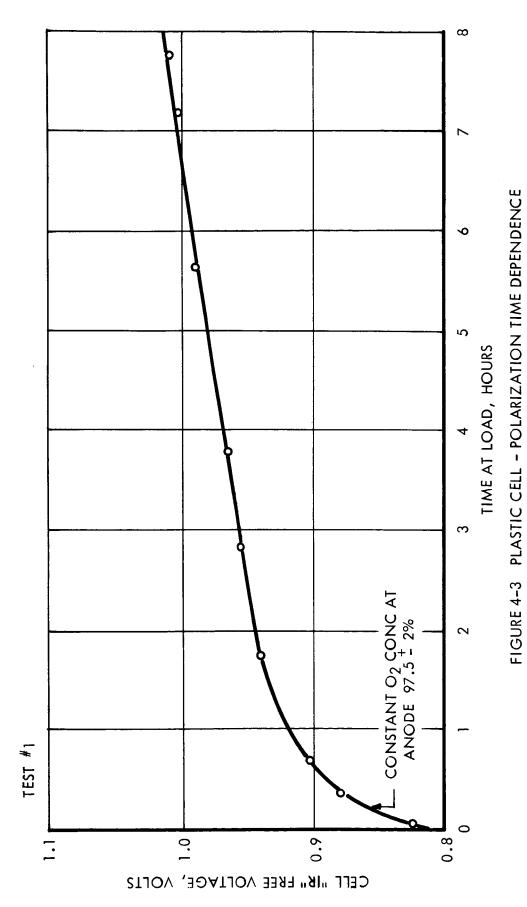


FIGURE 4-2 SMALL TEST CELLS



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4.2.3.3 Gold Plated Magnesium Cell - Additional extensive testing on the small cells will be accomplished using the gold plated magnesium cell which is now in the process of being gold plated (2 mil thick).

5.0 HARDWARE FABRICATION

5.1 Large Experimental Cells

Machining of all pieces required for the first two stage cells plus two spare cells has been completed. Pieces for two stage I cells and two Stage II cells are now in the process of being gold plated. The spare stage I and II cells will be sent out for application of gold plate upon receipt of initial four cells.

No unusual problems were encountered in machining the large cell end plates. After machining of the first cell end plate, a change in the design of the lightening holes was made to speed up the task of machining the remaining eleven end plates for the Stage I and II cells. The first plate used rectangular lightening holes, while subsequent plates used round drilled lightening holes.

Pieces for the Stage III acid cells are now under design. Fabrication will proceed upon design approval.

5.2 Test Stand

Fabrication and assembly of all components required for test stand operation has been completed. Minor modifications in plumbing and control circuits are being accomplished as individual functions of the test stand are checked for proper operation. Module assembly will proceed as plated cells are received.

6.0 OBJECTIVES

It is anticipated that the following objectives will be attained during the next reporting period.

- 1. All cells and spares, including third stage, will be completely fabricated and readied for testing program.
- 2. The large cell test stand will be put into full operation.
- 3. Short term parametric tests on all three cell stages will be completed.
- 4. The detailed analyses of the thermal characteristics of the three cell stages will be completed.
- 5. Operation of a gold plated magnesium acid stage cell will be tested in the small cell test rig.
- 6. Operating conditions for the three-stage life test will be recommended to NASA.

7.0 CONCLUSIONS

At the present date no valid conclusions regarding the Carbon Dioxide Concentration System or cells can be made without experiment data obtained from the large cells.

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